

Protocol for Atomic Oxygen Testing of Materials in Ground-Based Facilities

Version Number 2

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ABSTRACT

A second version of standard guidelines is proposed for improving materials testing in ground-based atomic oxygen environments for the purpose of predicting the durability of the tested materials in low Earth orbit (LEO). Accompanying these guidelines are background information and notes about testing. Both the guidelines and the additional information are intended to aid users who wish to evaluate the potential hazard of atomic oxygen in LEO to a candidate space component without actually flying the component in space, and to provide a framework for more consistent atomic oxygen testing in the future.

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EXECUTIVE SUMMARY

A goal of the Ballistic Missile Defense Organization's (BMDO's) Space Environment and Effects (SEE) Program is to develop tools that will help the space user community assess the risk to candidate spacecraft components arising from various effects of the low Earth orbit (LEO) environment. An important tool is a protocol for ground-based atomic oxygen testing. This protocol is intended 1) to provide a framework for more consistent testing, so that tests conducted at different times or in different facilities can be compared in a meaningful way (i.e., to create a more reliable database) and 2) to improve predictions of materials durability in LEO.

The protocol contained herein represents the first revision of the original protocol, which is dated April 1, 1994. The revision is based on new ground-based testing conducted by the author and by other facility operators who received contracts from the BMDO SEE Program to perform materials exposures in atomic oxygen environments. Additional work intended to lead to further refinement of the protocol is currently being implemented.

In keeping with the original intent, this revised protocol addresses only the atomic oxygen test itself, i.e., it describes how to ensure a reliable test with known and valid test parameters, and not the analysis of the tested samples. The key elements of the current protocol remain unchanged:

- Facility qualification - a document generated by the facility operator which verifies the level of facility qualification and aids the user in choosing a facility for a test.
- Test procedures - standardize testing.
- Specification of test parameters - informs user and facility operator which parameters shall be controlled.
- Report requirements - provide for a standardized written record of test parameters.

In this revised version of the protocol, three classification levels for testing have been defined. These levels are based on fundamental differences in the interaction mechanisms of materials with various exposure environments. With the definition of classification levels come more concrete guidelines for facility qualification and for control of the exposure environment. The new knowledge and test considerations that have led to the definition of these classification levels should give users enhanced confidence in the predictive ability of a given test.

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PREFACE TO VERSION NO. 2

This second version of the "Protocol for Atomic Oxygen Testing of Materials in Ground-Based Facilities" is incrementally revised over the first version. The objective and basic form remain unchanged. However, new knowledge that has been generated by the Ballistic Missile Defense Organization (BMDO) Space Environment and Effects (SEE) Program has permitted more precise definition of test environments, and discussions with Program participants have provided inspiration for the classification of facilities and tests. The introduction of classification levels represents the most substantial revision of the protocol.

CLASSIFICATION LEVELS OF TESTING

Atomic oxygen test environments differ fundamentally, and confusion still surrounds the validity of a test. In order to improve confidence in testing and to develop a common language for describing testing, classification levels for testing have been defined and added to this protocol. Three classification levels have been defined in Section 3 of this protocol. They are based on fundamental differences between the interactions of material surfaces with ground-based exposure environments. Briefly, Level 1 refers to screening tests, Level 2 refers to testing in an O-atom environment representative of a space flight in low Earth orbit (LEO), and Level 3 refers to testing for synergistic effects of atomic oxygen in combination with ions and/or VUV light. Both the degree of qualification of a test facility and the nature of the test itself can be described in terms of these three classification levels. Two key advantages of classification levels for atomic oxygen testing are listed below:

- Basis for comparison of tests
 - common language to describe tests quickly
 - eases the confusion about different facilities and tests
- Recognizes the differences between facilities and tests
 - makes clear that some tests are more representative of space than other tests
 - clarifies tradeoffs of choosing different types of tests
 - clarifies facility qualification

RATIONALE FOR THREE CLASSIFICATION LEVELS

An ideal test would involve exposure of a material sample to an environment that perfectly simulates the expected environment of the material in LEO for the duration that the material will be useful to the planned mission. However, the difficulty of producing fast atomic oxygen in the laboratory and the need for accelerated testing require that compromises be found.

The simplest way to test the oxidation resistance of a material is to expose it to an oxygen plasma or to the flowing afterglow of a plasma. The problem with such simple testing is that the interactions taking place at the surface of the material are fundamentally different from those that would occur in space. The different nature of the interactions is manifested in both resultant surface chemistry and topography. For example, work done by the author (unpublished) as part of the BMDO SEE Program has shown clear differences between the X-ray photoelectron spectra (XPS) of Kapton surfaces exposed to an oxygen plasma and Kapton surfaces exposed in space, suggesting different oxygen-carbon bonding on the surface. In addition, surfaces exposed in an environment where atomic oxygen impingement is largely isotropic, such as a plasma or flowing afterglow, become less roughened than surfaces exposed in LEO. While the interaction mechanisms differ from space, plasmas and other environments that produce low-translational-energy O atoms may still be useful for a quick and inexpensive assessment of the relative oxidation resistance of a candidate spacecraft material. Such a quick assessment, or screening, would constitute the lowest level of testing and is designated as "Level 1 testing." Predictions about the performance of a material in LEO that are based on Level 1 testing should be treated with caution because such testing is far from representing the space environment.

A higher level of testing would be done in an exposure environment that does induce material degradation through interaction mechanisms that are representative of those in LEO. Such environments are much more difficult and costly to produce than plasma environments. They typically involve a means to generate atomic oxygen and accelerate the O atoms to hyperthermal velocities in a directed beam. As noted previously (see "Preface to Version No. 1"), there may be many by-products in the exposure environment that could act synergistically with atomic oxygen to produce degradation that is greatly enhanced over O atoms alone. The most important of these by-products are VUV light and ions. The space environment also contains charged particles and VUV light, and the combination of atomic oxygen, VUV light, and ions to which a spacecraft material is exposed will depend strongly on the mission profile. Therefore, a user may wish to test a candidate material in an environment where the interaction is dominated by atomic oxygen or in an environment where VUV light and/or ions play a role in the surface interactions. Because the interaction mechanisms of O atoms reacting in combination with VUV light or ions are likely to be significantly different from those when O atoms are reacting alone, two classification levels have been defined for environments that are considered to be representative of space -- Level 2, which does not include synergistic effects, and Level 3, which does. A Level 3 test may take advantage of inherent VUV light or ions in the exposure environment, as long as their levels can be characterized, reproduced, and, possibly, controlled. For a Level 2 test, the environment must contain very little inherent VUV light and ions, or these by-products must be prevented from interacting with the test sample.

The choice of which test level to use for a high fluence ($\geq 10^{21}$ O atoms/cm²) test is not easy. High fluence tests are difficult and costly, so most users will want to extrapolate. The validity of extrapolating to very high fluences is still open to question, but extrapolation may be the only option. Users may be tempted to have a high-fluence test done in a plasma environment, where a high Kapton-equivalent fluence can be reached more easily than in a beam environment. However, there is little reason to believe that such a high fluence test will have

any better predictive ability than extrapolating from a lower fluence beam test. It is likely to be worse. The fundamental interaction mechanisms in a plasma involve thermal (kinetic energy) atoms and molecules, high-energy ions, VUV light, excited-state neutrals, and isotropic impingement (of reactive oxygen). Therefore, the degradation of a material in a plasma will be different than in a beam. A high-fluence test will only accentuate the differences.

The detailed interaction mechanisms of atomic oxygen with materials are still poorly understood; therefore, the most reliable testing will be that which best simulates the expected space environment. In fact, the details of the interaction mechanisms likely differ from material to material. We have the basic knowledge to create classification levels but not to predict space performance based on any type of test. We must still rely on testing that is most representative of the expected space environment. The classification levels for testing provide the user guidance in achieving a test in which (s)he can be confident as suiting her/his needs.

CLASSIFICATION CRITERIA

The criteria by which the classification levels are defined have been selected through consideration of many factors, which are enumerated as follows:

- **O-atom translational energy.** O atoms reacting at thermal energies induce surface chemistries on polymers that are different than those produced with O atoms reacting in space or in hyperthermal beam facilities ($E_T \sim 5$ eV). Also, the erosion rate of a polymer is many orders of magnitude slower with thermal O atoms than with hyperthermal O atoms. Therefore, there must be a qualitative difference between the chemical interaction at low and high translational energies. The question of where to draw the line in defining a classification level is nontrivial. Under contract with the BMDO SEE Program, the erosion of identical samples of an amorphous carbon film was studied in two exposure environments, one with an average O-atom translational energy of 2.2 eV and the other with an average O-atom translational energy of 5 eV. In both cases, the material loss rate was monitored with a quartz crystal microbalance (QCM) and the total test fluence was determined by erosion of a Kapton standard. The Kapton-equivalent erosion yield for the carbon was the same within 30% in both environments. It is reasonable to assume that while the erosion rate might be different at the two energies, the erosion mechanism must be similar because the Kapton and carbon samples eroded in approximately the same ratio. This result suggests that O atoms with translational energies even as low as 2.2 eV are likely to produce the same type of degradation seen on materials in LEO. Other studies have suggested that there is a steep rise in the erosion yields of several materials as the O-atom translational energy increases from one to two electron volts.^{1,2} Above two electron volts, the dependence of erosion yield on translational energy becomes weaker. Therefore, a reasonable lower limit of 1.5 eV for testing in a LEO-like environment (Levels 2 and 3) has been chosen. The upper limit on the average O-atom translational energy has been chosen to be 6 eV, which is within range of O-atom impact energies in LEO but should be below the energy

required for significant physical sputtering or atom displacement in the solid. While it is possible that atom displacement or sputtering might not be important at collision energies below 10 eV, there could be a high energy tail in the O-atom translational energy distribution that could extend several electron volts above the average energy. Hence, 6 eV is a reasonable upper limit for the average energy.

- **VUV light.** Recent data from the EOIM-3 space-flight experiment imply that the addition to the exposure of VUV light at a flux of one equivalent sun only alters the erosion yield of a VUV-sensitive polymer (polymethylmethacrylate) by about 20 percent.³ This level of uncertainty is similar to the uncertainty in an atomic oxygen test fluence that is based on Kapton erosion. In most cases, then, a VUV light flux of one equivalent sun will probably have little or no effect on the outcome of an atomic oxygen test. Thus, a concomitant VUV flux of one equivalent sun or less is considered to have no significant synergistic effect and is acceptable for Level 2 testing. It is possible that the ratio of VUV light flux to atomic oxygen flux might affect a material degradation rate. One equivalent sun of VUV exposure might therefore induce a larger synergistic effect with decreasing atomic oxygen flux. Because most ground-based testing is done in an accelerated mode with high fluxes of atomic oxygen, such testing might tend to underestimate the synergistic effects of VUV light in LEO even when the VUV exposure flux is one equivalent sun. However, at this time, the uncertainty caused by differences in the ratio of VUV light and atomic oxygen fluxes is judged to be small, and no specification is given in this protocol. Atomic oxygen testing with a VUV light flux higher than one equivalent sun may show significant synergistic effects at any O-atom flux and is therefore classified as Level 3 testing.
- **Ions.** It has been pointed out⁴ that the erosion yield of FEP Teflon is especially sensitive to the mole fraction of ionic oxygen (O^+) in an exposure environment where hyperthermal oxygen atoms are the dominant reactive species. Assuming no dependence of the material degradation rate on the ratio of ion flux to O-atom flux, a mole fraction of 0.01 appears to enhance the erosion yield of FEP Teflon by one or more orders of magnitude over the erosion yield expected if no ions were present. Most, if not all, other polymers appear to have erosion yields that are almost independent of an ionic mole fraction in the range 0.01 or less. However, there is a chance that a candidate spacecraft material might show a synergistic effect as significant as that seen in the erosion of FEP Teflon. In this case, a test with a significant ion fluence would lead to an overly pessimistic prediction about the degradation rate of the material in very low Earth orbit (say less than 300 km), where the mole fraction of ions is approximately 10^{-4} . Allowing for the possibility of ion-sensitive candidate materials, the upper limit in ion exposure for the non-synergistic (Level 2) classification of testing has been assigned as 10^{-4} of the effective O-atom fluence, which is the lowest ion fraction that would typically be encountered in LEO. Even if a spacecraft is expected to fly at very low altitudes where the ion flux will be insignificant, a Level 3 test (ion fraction greater than 10^{-4}) may still be satisfactory because it should provide an upper limit on the amount of degradation predicted for a candidate material.

- **Directionality of atomic oxygen impingement.** Given the nominal orbital velocity of a spacecraft in LEO and the mean speed of oxygen atoms at an ambient temperature of 1000 K, the angular range of O-atom attack at any point on a surface will be on the order of 20 degrees or less. The directionality of attack is important in the development of surface topography during erosion. Highly-directional O-atom impingement on erodible materials leads to rougher surfaces than isotropic impingement. This roughening may affect optical and thermal properties of a material. Therefore, Level 2 and 3 testing require that the direction of impinging atomic oxygen vary by no more than 20 degrees at any point on the sample surface.

KAPTON AS A TEST-FLUENCE STANDARD

Kapton HN still endures in this second version of the protocol as a test-fluence standard. As mentioned above, the use of Kapton to determine the fluence in a test of the erosion yield of identical amorphous carbon samples in two different facilities leads to similar results for the erosion yield of the carbon. Furthermore, Kapton seems to be relatively insensitive to synergistic effects of O atoms with VUV light (~ 1 equivalent sun) and ions (mole fraction ≤ 0.01). There are many anecdotal indications that even higher levels of VUV light and ions would not invalidate the use of Kapton as a test-fluence standard. It is recommended here that a Kapton witness sample be used in every test, no matter what the classification level. For a Level 3 test that involves high fluxes of VUV light or ions, the user should consider also using a witness sample of standard amorphous carbon (see Section 4.6). If the Kapton-equivalent fluence leads to an apparent carbon erosion yield in the range 2×10^{-25} to 6×10^{-25} cm³/atom, then the Kapton-equivalent fluence should be used as a measure of the test fluence. If the carbon erosion yield falls outside this range, then the carbon erosion rate that has been calibrated against Kapton in the absence of significant synergistic effects should be used to determine the test fluence.

A COMMENT FROM THE AUTHOR

Based on my own experiences as a user and on the lack of feedback I've received, Version 1 of this protocol has had little effect on testing. Even when I specifically requested testing according to the protocol, the protocol was not followed. In my experience (and in the experience of other users who have talked with me), facility operators still appear to dominate the way testing is conducted. I have come to expect that "business as usual" will mean haphazard testing, with each facility operator defining his own set of guidelines for each test. No standard approach exists and it will not exist until users insist on it. As far as I can tell, users still feel the need to survey various facilities, going over much of the same ground that has led to this protocol, and they end up contracting with the facility operator that makes the best claim for a facility. This protocol, while undoubtedly imperfect, is the only standard set of guidelines for atomic oxygen testing that I know of, and it has grown out of my experience as an operator of a hyperthermal O-atom beam apparatus and as a user of other facilities for

testing. I have taken seriously the task of preparing these guidelines, and I would welcome constructive feedback on how they could be improved. Even in its current form, this protocol should be able to guide users to a reliable test. Through use and feedback, it could be refined further until everyone, both users and operators alike, can be comfortable with it. Improved consistency and reliability from ground-based atomic oxygen testing will only be achieved if users and facility operators make an effort to establish and follow a standard set of guidelines. I believe this protocol can serve as a firm foundation for such a test standard.

PREFACE TO VERSION NO. 1

OBJECTIVE

The objective of this protocol is to provide guidelines for materials testing in ground-based atomic oxygen environments for the purpose of predicting the durability of the tested materials in low Earth orbit (LEO).

BACKGROUND

Atomic oxygen in low Earth orbit (200-700 km), combined with high orbital velocities, gives rise to hyperthermal oxygen atom reactions on satellite surfaces. A typical O-atom number density at space shuttle altitudes is on the order of 10^8 cm^{-3} . An orbiting body traveling at 7.8 km/s through this density experiences a flux of $\sim 10^{14} \text{ O atoms/cm}^2/\text{s}$. The high velocity atomic oxygen impacts correspond to collision energies near 5 eV, and the $\sim 1000 \text{ K}$ ambient temperature gives a full width at half maximum (FWHM) energy spread of 3.9-7.0 eV to the collisions. The atomic oxygen concentration is dependent on many factors besides altitude, including solar activity, season, and variations in the Earth's magnetic field, latitude, and local time.^{5,6} Model calculations (e.g., those of MSIS-86^{6,7}) are usually used to calculate atomic oxygen number densities encountered for a particular mission.

Atomic oxygen is one of the most important hazards of the LEO natural space environment. Hyperthermal O-atom reactions can degrade materials through oxidation and erosion. The detrimental effect of atomic oxygen in LEO was first recognized after post-flight analyses of polymer and paint surfaces which were exposed during early space shuttle flights (STS-1, STS-2, STS-3).⁸ Polymers showed a loss of surface gloss and concomitant weight loss, while paint surfaces exhibited premature aging. Concern over the degradation of materials by atomic oxygen sparked a huge effort, involving space- and ground-based studies, that has been aimed at the identification, understanding, and solution of problems caused by O atoms in LEO.⁹⁻¹¹ Although the database is now voluminous, a detailed understanding of the interaction mechanisms has remained elusive.

Controlled experiments are difficult to perform in space, and sources of hyperthermal O atoms for ground-based studies are problematic. Most data therefore tend to be phenomenological, and the approach of the user community has generally been to test candidate materials in space and/or in ground-based atomic oxygen facilities and carry out functional analyses to assess any changes in material properties. Reliable ground-based materials evaluations are sought after as an economical alternative to space experiments. However, because of differences in atomic oxygen generation methods and test procedures, agreement between different test sites is rare. A profound problem is the inherent difficulty of making neutral, ground-state, hyperthermal O atoms in the laboratory. Some deal with this problem by producing low kinetic energy O atoms; many use plasma asheres; others create ions of atomic and

molecular oxygen. All sources of hyperthermal O atoms have as by-products one or more of the following: ultraviolet (UV) and vacuum ultraviolet (VUV) light, ions, excited-state species, and fast neutrals, such as O₂ and rare gas atoms. These by-products may affect apparent reaction rates by acting either alone or in combination with O atoms to degrade a sample.

The need for reliable testing still endures. Testing so far has not followed a set of standard procedures. A user desiring an atomic oxygen test of a candidate space material has been faced with a choice between as many test methods as there are facilities. Even though highly skilled researchers may perform the tests, differences in their methods compromise the validity of a test. In addition, the correlation between ground- and space-based test results has only been studied for a handful of materials. The user has had no recourse but to rely upon the experience and skill of the facility operator in order to obtain meaningful test results from a ground-based atomic oxygen facility.

Even though gaps exist in our knowledge about hyperthermal O-atom interactions, a systematic approach to testing that is, to the extent possible, independent of the test facility, would increase the reliability of ground-based tests and confidence in them. Recently, Banks et al.¹² have produced a document that attempts to standardize the atomic oxygen testing of protected polymers with the use of thermal plasma systems. This document is the first of its kind and is a valuable starting point for the development of more comprehensive test procedures. The test protocol contained herein attempts to be more comprehensive in that it is intended to be applied to all facility types. It is an expansion of the document by Banks et al. and not a rival set of procedures. In fact, recommendations by Banks et al. are included as part of this protocol.

Two recent tasks of the Ballistic Missile Defense Organization (BMDO) Space Environment and Effects (SEE) Program are other important sources of information for this protocol. In one task, 19 experimenters provided 6 identical samples of each of their contributed materials; 78 different materials were represented. One set of 78 material samples was flown on a passive exposure tray on the EOIM-3 experiment aboard the space shuttle Atlantis in August 1992 and exposed to ram attack from atomic oxygen for ~40 hours. A second identical set of samples was exposed to the same fluence of atomic oxygen in a ground-based facility. A third set of samples served as controls, and the remaining three sets were stored as spares. Analyses of the flight, ground, and control samples were carried out by investigators at JPL and by the sample providers. The results are contained in a report to BMDO.¹³ This task provided a direct comparison between the effects of hyperthermal atomic oxygen in a ground-based facility and in LEO on a variety of materials. In a second task, identical sets of four materials (materials that have been well-characterized in LEO) were exposed to the O-atom environments of five different types of atomic oxygen test facilities. Various exposure conditions for each material were specified with a goal of determining the important parameters that must be controlled in a ground-based test in order to produce a reliable prediction of longevity in LEO. While control of specified test parameters is often difficult, as well as variable from facility to facility, the results that were obtained and the experience gained from approaching the problem of testing as a user have afforded an important perspective in the preparation of this protocol.

The atomic oxygen literature and, especially, discussions with members of the space environmental effects community are also vital to the development of an atomic oxygen test protocol. This protocol is a first attempt to bring together in one place a comprehensive set of test procedures. Many of the procedures described here are already in practice at one or more test facilities. Nevertheless, the fact remains that a consensus is lacking in the methods by which atomic oxygen testing should be performed. Through more knowledge gained from additional research and through feedback from the space environment and effects community, this protocol will be revised and refined and perhaps grow into a widely accepted standard. In fact, the BMDO SEE Program includes specific follow-on tasks involving ground- and space-based testing that are aimed at the refinement of this protocol. At present, it is hoped that this protocol will aid users who wish to evaluate the potential hazard of atomic oxygen in LEO to a candidate space component without actually flying a material in space, and provide a framework for more consistent atomic oxygen testing.

EXPOSURE ENVIRONMENTS

Many types of facilities have been set up for ground-based atomic oxygen testing of materials.¹⁴ The sources that control the exposure environment in these facilities can be grouped broadly into thermal plasmas and directed beams. Both plasmas and nominally neutral, O-atom-beam sources can be considered for atomic oxygen testing. Facilities that subject test samples only to an ion beam are not considered in this protocol as the interactions of ions with materials are qualitatively different from interactions involving neutral O atoms, which are the active species in LEO. Plasma systems have an ion component, but they are considered because of their relatively low cost.

O-atom beam sources can be thermal or hyperthermal. A thermal source generates O atoms with a Maxwell-Boltzmann distribution of velocities at temperatures near 300 K. The average O-atom kinetic energy at this temperature is 0.04 eV, and the energy range is relatively broad (FWHM $\sim$ 0.06 eV). A hyperthermal source produces O atoms with average kinetic energies in excess of 300 K. Hyperthermal O atoms can be produced in a variety of ways, and the resulting velocity distribution can be Maxwell-Boltzmann, or it can be much narrower. The roughly 5 eV O-atom collision energy encountered on materials in LEO has motivated the development and use of hyperthermal sources, although many of these sources are routinely run with O-atom kinetic energies much less than 5 eV. Generally, in a facility that employs a beam source, a test sample is placed in a vacuum chamber where a nominally neutral beam is directed at it. A plasma frequently serves as the source of O atoms, so the exposure environment may contain unwanted but unavoidable by-products, such as residual ions, excited-state neutrals, and UV/VUV light. Beams may also contain molecular oxygen and inert carrier gases.

A sample can be placed directly into an oxygen or air plasma, in which case the application would be referred to as a plasma asher, or it can be placed downstream of an oxygen-containing plasma in the "flowing afterglow." In either arrangement, the O-atom kinetic energies are near thermal and impingement on the test surface is isotropic. A common radio

frequency plasma asher uses a 13.56 MHz capacitively-coupled air or oxygen plasma. A hybrid beam/plasma environment can be generated with an electron cyclotron resonance (ECR) plasma that uses 2.45 GHz excitation in a magnetic field to produce a somewhat directed beam of atoms and ions. In one facility,¹² the output of an ECR source is sometimes deflected from a quartz surface in order to reduce the exposure of the sample to ions and VUV light; however, all directionality of the O atoms is lost. Of these less-directional exposure methods, the flowing afterglow and deflected ECR beam give the exposure conditions with fewest by-products. Their environments consist predominantly of ground-state, thermal O atoms and other neutrals. The plasma environments subject test materials to intense UV/VUV radiation and high-energy ions (tens to hundreds of electron volts), in addition to excited-state neutrals and O atoms, whose kinetic energies are thermal.

Hyperthermal beams are more likely than thermal systems to induce degradation through reaction mechanisms that are the same as those in LEO. The reactivities of thermal O atoms with hydrocarbon polymers are three to four orders of magnitude lower than those observed in LEO or in hyperthermal beam facilities. And plasma ashers are well known to give widely varying results depending on operating conditions. On the other hand, hyperthermal beam systems are much more expensive to set up and operate than thermal-atom systems. The setup cost alone can be more than ten times higher for a beam apparatus than for a thermal-atom system. Therefore, thermal-atom testing may serve as a cost-effective screening method for the evaluation of the durability of a material in an atomic oxygen environment. It must be emphasized, however, that appropriate care must be taken in the interpretation of test results from thermal systems. Potential users of thermal-atom systems are referred to studies by Banks et al.⁸ and Koontz et al.¹⁵ for a thorough discussion of test parameters.

The interaction environment can also be better simulated in a beam system, if the operational pressure is low enough ($\leq 10^{-4}$ Torr). Atomic-oxygen-induced materials degradation occurs on orbit when an ambient oxygen atom impacts a surface and either forms a bond with another atom on the surface or breaks a bond, which leads to removal of one or more atoms from the surface. The atom-surface interaction occurs in the free molecular flow regime so each atom has one and only one chance to react. Hyperthermal beams operating at or near the space-vacuum level effectively simulate the interaction environment; therefore, when the flux or fluence of the impinging atoms is measured, the reaction probability or erosion yield can be computed in a straightforward manner. On the other hand, a source that operates in the transition or viscous flow regime (even if it is a beam source) can make meaningful calculation of the erosion yield difficult. In such an environment, measurement of the atom concentration is not sufficient, because a single atom can interact with the surface many times due to gas phase scattering. For sources that operate in this high-pressure mode, the collision frequency with the surface must be calculated in order to obtain the true erosion yield. This calculation requires detailed knowledge of the kinetic energy distribution of the atoms in the flow and the bulk gas temperature. Although an individual beam system can produce a consistent set of exposure conditions from test to test, each beam facility type offers a distinct environment for testing. Key variables include average O-atom kinetic energy, the width of the energy distribution, atomic oxygen flux, molecular oxygen flux, inert gas flux, ion flux, excited-state neutral flux,

UV/VUV flux, radiative heat load on the sample, sample temperature, chamber pressure, and nature of background gases.

There is a broad consensus that atomic oxygen oxidizes and degrades materials in LEO via chemical reaction mechanisms similar to those that occur in combustion systems on the ground; however, the dependency of the reaction probability (or erosion yield) on the various exposure parameters is a subject of much discussion. Therefore, the extent to which these parameters must be controlled or reported remains uncertain. A notable example is O-atom kinetic energy. Some researchers have reported an energy threshold for Kapton erosion above which the O-atom reactivity becomes independent of energy.¹ In contrast, others report a strong energy dependence for several materials, including Kapton, when they are exposed to atomic oxygen kinetic energies between 1 and 12 eV.^{2,16} Such discrepancies make the definition of test standards difficult. Moreover, even if the interaction mechanism for a particular material were well understood, the behavior of a new candidate material is likely to be completely different. Given the uncertainties at present, the user would wish to choose the facility that most closely simulates the LEO environment. However, no single facility can truly simulate LEO, and no facility can produce results that agree with space data for all types of materials. The approach to testing, then, must be phenomenological. Regardless of the facility chosen for a test, the validity of the test must rest on the proven ability of the facility to induce, in a variety of materials, effects that can be related in a straightforward way to the effects of the LEO environment. Because of lingering uncertainties about interaction mechanisms, the requirements that are placed by this protocol on exposure parameters are fairly liberal. Nevertheless, if future atomic oxygen testing is to provide consistent and useful results, as well as improved test methods, then these requirements should be followed.

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RECOMMENDED PRACTICES

1. SCOPE

This protocol comprises standard procedures for the testing of materials in an atomic oxygen environment. Issues associated with such a test that are covered in this protocol include facility requirements, sample handling, exposure conditions, verification of exposure conditions, and reporting of results. While facility qualification levels are discussed, no specific facilities are recommended. Within the context of verification of exposure conditions, standard methods for the measurement of erosion and surface contamination are given. Because facility capabilities and user needs vary and uncertainties in procedures still persist, many recommendations have associated notes to inform the user of various test considerations.

The desired atomic oxygen exposure area and the volatility of the test material limit the variety of samples that can be exposed. Certain facilities can accommodate sample areas of 50 cm² or more, while others are limited to less than 1 cm². Typical samples are 1.25-2.54 cm in diameter because these dimensions are common in space-based tests, but the sample dimensions for a given ground-based test could vary widely depending on the test facility. The exposure is conducted in a vacuum chamber, so the material must not outgas enough to affect the evacuation of the test chamber. Materials intended for use in space typically have low outgassing characteristics, therefore outgassing in a ground-based test is only a consideration insofar as it may cause contamination of the test environment.

This protocol is not intended to address the myriad analyses that often follow an atomic oxygen test in order to determine the extent of degradation of a given material; however, two straightforward methods for determining erosion yield are provided in the context of determining the O-atom fluence of a test from the erosion of a witness sample, and these methods may be applied to the evaluation of actual test samples if applicable. Nevertheless, the evaluation and criteria used for acceptance or rejection of a tested material shall be the responsibility of the user. Characterization of any functional changes in the tested material is vital to ensure the suitability of the material for use in LEO. In some cases, the functional tests may have to be done *in situ* during the atomic oxygen exposure.

2. APPROACH

The individual, or user, who desires to evaluate materials in an atomic oxygen test facility ultimately defines the specific test parameters based on the unique requirements of a test as well as the capabilities of the facility chosen for the test. The operators of an atomic oxygen test facility shall assist potential users by preparing a facility qualification document that describes their facility, its capabilities, and the methods by which exposure parameters are determined. With such a document and this protocol, a user can make an informed decision about where and how a test is to be conducted.

While this protocol applies to testing in facilities employing either thermal or hyperthermal sources of oxygen atoms, the "thermal-atom" systems (e.g., plasma ashers and flowing afterglows) should only be regarded as screening tools. Plasmas and other exposure environments that contain near-thermal O atoms and/or ions of any velocity can give an indication of the resistance of a material to atomic oxygen attack, but the hyperthermal-atom sources, which consist of a directed beam of high-velocity oxygen atoms, produce effects that are easier to correlate with those expected in the LEO environment. Therefore, it is recommended that a beam source of hyperthermal oxygen atoms be used for any test from which a reliable prediction of performance in LEO is required.

Because of the large variety of test facilities, three classification levels are defined for testing. These levels are designated Level 1, Level 2, and Level 3, and they serve to categorize the qualification level of a test facility, as well as the test itself. In general terms, Level 1 refers to screening in a plasma or other environment which poorly simulates hyperthermal O-atom attack in space; Level 2 refers to testing in a hyperthermal O-atom beam in the absence of complicating effects arising from a synergistic interaction of O atoms with VUV light or ions; and Level 3 refers to testing in a hyperthermal O-atom beam with the addition of known fluxes of VUV light and/or ions. Level 3 is divided into three sublevels to indicate if one or more synergistic agents are present: VUV light, ions, or both. The facility qualification document will allow the user to determine the level of testing capable at a given facility.

3. TERMINOLOGY

3.1 Classification levels. Categories of testing that are based on fundamental differences between the interactions of surfaces with exposure environments.

3.2 Contamination witness. A material specimen, whose surface is known not to erode in the LEO environment (e.g., MgF_2 , SiO_2 , germanium-coated Kapton), that is placed in a test-sample position and subjected to atomic oxygen attack and monitored (either *in situ* or *ex situ*) for the buildup of contaminants arising from the exposure environment.

3.3 Control sample. A specimen that is identical to either the test sample (test control) or witness sample (witness control), is not exposed to direct O-atom attack in the harsh O-atom exposure environment, and is used as a standard of comparison in judging exposure effects.

3.4 Erosion. Removal of matter from a material.

3.5 Erosion yield. Amount of matter removed. Usually expressed as either volume per unit area or mass per unit area.

3.6 Facility operator. Individual who operates a ground-based atomic oxygen test facility and who conducts a test by exposing materials to a simulated LEO atomic oxygen environment.

3.7 Level 1 test. A test in which the interaction mechanisms of the exposure environment with a surface are fundamentally different from the interaction mechanisms of atomic oxygen with spacecraft surfaces in LEO, *with or without the presence of VUV light or ions*. In a Level 1 test, the surface chemistry and/or topography of the exposed material are/is not representative of exposure of the same material in LEO. Level 1 test environments must contain oxygen and include plasma systems, flowing afterglows, effusive and other low-translational-energy ($E_T < 1.5$ eV for atomic oxygen) beams, and any other environment that exposes a test surface to substantial ion bombardment, low-translational-energy reactive oxygen, or isotropic impingement of neutral reactive species.

3.8 Level 2 test. A test in which the interaction mechanisms of the exposure environment with a surface are fundamentally similar to the interaction mechanisms of atomic oxygen with spacecraft surfaces in LEO, *in the absence of VUV light and ions*. The VUV light flux in a Level 2 test is ≤ 1.0 VUV suns. The ion fluence of a Level 2 test is $\leq 10^{-4}$ of the Kapton-equivalent atomic oxygen fluence. The average translational energy of impinging oxygen atoms in a Level 2 test is in the range 1.5 to 6.0 eV, and may vary within this range during the test. The incoming direction of the impinging atomic oxygen in a Level 2 test must be within ± 10 degrees of the nominal direction over the entire sample surface. A typical Level 2 test involves a beam that contains atomic oxygen and has very low inherent VUV light and ionic content. A Level 2 test could be performed with any atomic oxygen source that produces O atoms with sufficient velocity and degree of collimation, as long as any significant VUV and/or ionic component can be blocked from the sample surface.

3.9 Level 3 test. A test in which the interaction mechanisms of the exposure environment with a surface are fundamentally similar to the interaction mechanisms of atomic oxygen, *in combination with VUV light and/or ions*, with spacecraft surfaces in LEO. A Level 3 test is a Level 2 test with the addition of a known fluence of VUV light or ions, to which the sample surface may be exposed simultaneously or sequentially with atomic oxygen. To differentiate between the various combinations of tests, the following sub-levels are defined:

- Level 3a: known flux of VUV light; ion fluence of test $\leq 10^{-4}$ of the Kapton-equivalent atomic oxygen fluence
- Level 3b: known fluence of ions; VUV light flux ≤ 1.0 sun
- Level 3c: known flux of VUV light and ions, with flux of each above the Level 2 limits

Unless specified by the addition of the letters "seq" (for sequential) after the classification sub-level, a Level 3 test shall be assumed to consist of simultaneous exposure of the test material to atomic oxygen and the relevant combined agent(s). (Any test that does not include atomic oxygen as part of the exposure is beyond the scope of this Protocol.)

3.10 Mass loss. Refers to removal of matter from a specimen. A negative mass loss indicates an increase of mass in/on the specimen.

3.11 Mass-loss measurement. Any measurement that can lead to the determination of the amount of matter lost or gained by a specimen.

3.12 Test. As defined by this protocol, the test includes all the steps necessary to ensure that a controlled and characterized exposure of materials to a simulated LEO atomic oxygen environment is performed, including the exposure step itself.

3.13 Test sample. A specimen of a material or component whose durability under atomic oxygen attack in LEO is to be inferred from a test.

3.14 User. Individual who desires to evaluate the resistance of a material or component to atomic oxygen attack in LEO by testing a sample of the material or component in a ground-based facility.

3.15 Weight loss. Refers to the reduction in weight of a sample that can be determined by weighing on a balance. A negative weight loss indicates a weight gain.

3.16 Weight-loss measurement. A mass loss measurement, but more specific as it is a measurement of a change in weight as determined by a balance.

3.17 Witness sample. A material specimen, Kapton HN or a material whose erosion rate relative to Kapton HN is known for a given set of exposure conditions, whose erosion yield is used to measure and verify the Kapton-equivalent atomic oxygen exposure fluence.

4. FACILITY QUALIFICATION

Operators of each atomic oxygen test facility shall prepare a document that describes their facility, and this document shall be updated if modifications to the facility are made or new knowledge becomes available that indicates the exposure parameters in the current document are no longer valid. Facilities differ in the level of testing achievable, and even within a given test level, exposure parameters and methods may differ from facility to facility. However, many restrictions apply which are key to conducting a valid test within a specified level. The user should ensure that the requirements for the desired test level are met before selecting a facility for testing. The information to be included in the facility qualification document is grouped into two categories: "Specifications" and "General information." This information, including the proof that the facility specifications are met, shall be required so as to ensure the user a valid test {1}.

Specifications

4.1 Atomic oxygen kinetic energy (applies to Level 2 and 3 facilities only). The range of available mean O-atom kinetic energies and concomitant energy distributions shall be reported. For the low and high limits in the mean energy reported, the mean energies and the energy distributions shall be measured directly (e.g., by time-of-flight methods or acceleration of an O ion in a known potential before charge neutralization). A mean kinetic energy that can

be maintained in the range 1.5 to 6.0 eV is necessary to qualify a facility for Level 2 or 3 testing, while a mean kinetic energy less than 1.5 eV shall only qualify a facility for Level 1 testing. Mean kinetic energies greater than 6.0 eV shall not be used.

4.2 Atomic oxygen flux. The range of available average atomic oxygen fluxes shall be reported. A qualified facility shall be capable of an average O-atom exposure flux that falls in the range 10^{14} - 10^{17} atoms/cm²/s. Experimental evidence shall be provided to show that at the extremes of the reported flux range, the flux remains constant to within 20 percent over the duration of a test with an O-atom fluence of 10^{20} atoms/cm². It shall be acceptable for any facility to derive the average flux from an erosion measurement of a Kapton HN standard, where the erosion yield is taken to be 3.00×10^{-24} cm³/atom at 5 eV and 25°C, and the density is 1.42 g/cm³. Other, more direct measurements of flux, such as the resistance change of a silver strip that is ≤ 50 nm thick, during exposure to atomic oxygen shall also be acceptable.

4.3 Exposure area. The largest exposure areas achievable with the minimum and maximum reported fluxes, respectively, shall be reported. The flux deviation over the reported areas shall not be more than 20 percent. This deviation shall be verified with the exposure of witness samples. A functional form for the flux distribution of the beam over the reported area is recommended.

4.4 Degree of collimation (applies to Level 2 and 3 sources only). The largest exposure area over which the O-atom impingement angle does not deviate by more than 10 degrees from the surface normal shall be reported. The angle may be determined by geometry if the atomic oxygen originates from an effective point source. Alternatively, the impingement angle may be determined from the angle of the conical features on the surface of eroded Kapton. The method used to determine the impingement angle shall be reported.

4.5 Figure of merit. As a figure of merit, the product of the average atomic oxygen flux I and the exposure area A shall be reported. The product IA that is reported shall be the highest value achievable under routine operating conditions. This value shall be defined for areas which satisfy both a flux deviation of ≤ 20 percent (Section 4.3) and an O-atom impingement angle of ≤ 10 degrees (Section 4.4). Kapton equivalent O-atom flux may be used to determine this value. Acceptable values shall be in the range 10^{13} - 10^{18} atoms/s.

4.6 Calibration. Facilities shall report the results of an erosion yield measurement of a carbon standard at 25°C. The only acceptable standard is a carbon film that is deposited onto a substrate by laser ablation in the laboratory of Prof. R. H. Prince at York University in Toronto, Ontario, Canada. [Department of Physics, York University, North York, Ontario, Canada, M3J 1P3; Tel. (416) 736-5051; e-mail: bob@unicaat.yorku.ca. (Request a standard amorphous carbon film for an atomic oxygen erosion study.)] With the use of a known Kapton-equivalent flux applied over a known exposure time and the carbon film density of 2.26 g/cm³, the erosion yield of the carbon standard must fall in the range 2×10^{-25} to 6×10^{-25} cm³/atom. Any mass-loss measurement shall be acceptable for determining the erosion yield of the carbon sample. The details of the test shall be reported according to the guidelines in Section 8.

4.7 Contamination. The nature and levels of contaminants expected from the O-atom source and from the vacuum chamber shall be reported. For source contamination, witness samples at the exposure position which yields the maximum figure of merit (Section 4.5) shall be used. For vacuum chamber contamination, witness samples at the typical position of chamber control samples shall be used. Contaminants may be identified and quantified by surface analysis (e.g., X-ray photoelectron spectroscopy) of the witness samples which are subjected to direct O-atom attack or placed in the chamber out of direct O-atom exposure {2}. Particulates can be characterized with scanning electron microscopy in conjunction with energy dispersive spectroscopy (EDS). In addition, the total rate of accumulation of contamination may be measured with a quartz crystal microbalance. A mass spectrometer may be used to determine species present in the source or chamber and therefore may be helpful in understanding the surface analysis or microbalance results. For each contaminant present, the operator shall provide the rate of accumulation on a specified test sample and how this rate was determined. The rate of accumulation of contaminants shall be reported in units of monolayers per 10^{20} O atoms/cm² (Kapton-equivalent fluence). Total contamination loading of a non-erodible sample shall not exceed 0.5 monolayers per 10^{20} O atoms/cm² {3}. The accumulation rate of particulate contamination shall be reported in units of particles per cm² per 10^{20} O atoms/cm², and the average particle size and size range shall also be reported. Exposures of 10^{20} O atoms/cm² shall cover less than 10 percent of the surface area with particles, and the mass increase caused by the accumulation of particles shall be less than 1 μ g {4}.

4.8 Species other than oxygen atoms (applies to Level 2 and 3 facilities only). The nature and flux of species other than O atoms that will impinge on a sample that is exposed at the position that yields the maximum figure of merit shall be reported along with the measurement method. A mass spectrum of the exposure environment may be provided in conjunction with this report. The O⁺ (and/or O⁻) and O(¹D) fluxes shall each be specified as a fraction of the 5 eV Kapton-equivalent O-atom flux and may also be reported as an absolute measurement. In systems employing a carrier gas, the total ion flux shall also be reported. If these quantities have not been measured directly, then an upper bound based on the sensitivity of the measurement shall be presented as a fraction of the O-atom flux. Qualification for Level 2 testing requires a facility operator to demonstrate that the facility is capable of exposures at a total ion flux that is less than or equal to 10^{-4} of the Kapton equivalent O-atom flux. Qualification for Level 3 testing requires a facility operator to demonstrate that the total ion flux for a typical test is known. An operator claiming the capability for Level 3 testing shall report the nature of the ions and the achievable range of fluxes, both as a fraction of Kapton-equivalent O-atom flux and as an absolute measurement.

4.9 UV/VUV spectrum and flux (applies to Level 2 and 3 facilities only). The UV/VUV spectrum, flux, and measurement method(s) shall be reported. The flux shall be the flux that impinges on a sample placed at the position which gives the figure of merit in Section 4.5. Flux shall be reported in "suns" (i.e., the ratio of the intensity at the sample at the Lyman alpha wavelength to that which is felt by a body in direct sunlight in LEO). Absolute units may also be given. Qualification for Level 2 testing requires a facility operator to demonstrate that the facility is capable of exposures at a VUV light flux that is less than or equal to 1.0

equivalent suns. Qualification for Level 3 testing requires a facility operator to demonstrate that the VUV flux for a typical test is known. An operator claiming the capability for Level 3 testing shall report the achievable range of VUV fluxes.

4.10 Common attributes. Verification shall be provided that the following attributes exist or are practiced at the facility.

4.10.1 Tools to permit minimum abrasion and contamination of samples during manipulation.

4.10.2 Ability to mount a witness sample where it is subjected to direct atomic oxygen attack such that it will experience the same atomic oxygen fluence as the test sample.

4.10.3 Ability to mount a number of control samples in the chamber, where they are removed from direct atomic oxygen attack {5}.

4.10.4 Ability to control and cycle the bulk temperature of all tested samples including the direct-exposure witness sample. The controllable temperature range shall at the minimum be from near ambient to 100°C.

4.10.5 The test chamber is operated in a manner to minimize contamination. In particular, the chamber is free of oils, such as residue from vacuum pump fluids.

4.10.6 No silicone-containing compounds, such as greases, oils, adhesives, or O-rings, are used anywhere outgassing or reaction products may enter the test chamber {6}.

4.10.7 All gases used in testing are ultra-high-purity grade ($\geq 99.98\%$). In any system employing carrier gases, only the rare gases, helium, neon, or argon, shall be used {7}.

General information

4.11 Maximum fluence. The maximum fluence that can be attained in a continuous test for the exposure area that was used to calculate the figure of merit *I*_A in (Section 4.5) shall be reported {8}. The maximum accumulation of atomic or molecular contaminants during a test shall not exceed 0.5 monolayers, and total obscuration resulting from particulates shall be less than 10 percent. Fluence is integrated flux and shall be reported in units of O atoms/cm².

4.12 Mounting methods. The method by which samples are mounted, O-atom impingement angles other than normal, and sample temperature control capabilities and methods shall be described. Masking methods provided by the facility for step-height measurements and for allowing exposed and unexposed areas of the sample to be compared shall also be reported.

4.13 Synergistic studies. Capabilities for studies of synergistic effects, e.g., UV/VUV light source, charged particle source, surface dosing with a simulated contaminant, thermal cycling, etc., shall be reported.

4.14 Available surface-analysis techniques. A description of available post-test surface-analysis techniques shall be reported.

4.15 Available *in situ* analysis techniques. A description of available *in situ* analysis techniques, e.g., quartz crystal microbalance, fiber optic sensor, current measurement for a silver actinometer, reaction product identification, X-ray photoelectron spectroscopy, absorbance, etc., shall be reported.

4.16 Mass-loss measurements. Capabilities to perform mass-loss measurements shall be reported. For facilities offering this service, provisions for both *in situ* (e.g., quartz crystal microbalances) and *ex situ* (e.g., pre-/post-weight loss or surface recession) measurements shall be reported. The precision and accuracy of each measurement method shall also be reported.

4.17 Ground-space correlation. A summary record may be provided which illustrates the correlation between the effects observed in the facility and those observed in LEO for all materials where such comparative data are available {9}.

4.18 Overview of the facility. A description of the O-atom source, the chamber, the vacuum system, pump-down procedures, pump-down time, available clean room/bench facilities, personnel, and any other relevant information shall be provided.

5. SAMPLE HANDLING

5.1 Minimization of abrasion. Samples shall be handled in a manner that will minimize abrasion to any part of a sample.

5.2 Minimization of contamination. All samples shall be handled indirectly with clean and noncontaminating instruments. Instruments shall be cleaned prior to use in the manner used to clean the samples.

5.3 Sample cleaning. The user is responsible for cleaning test samples prior to delivery to the test facility. Additional cleaning at the facility shall be arranged with the facility operator. Witness samples may be cleaned by rinsing with an organic solvent or solvent mixture that will not dissolve the sample material and that has a nonvolatile residue of less than 10 ppm. Dust may be blown off samples (or containers) with an ultra-high-purity inert gas (including nitrogen from a liquid nitrogen source) {10}.

5.4 Sample storage. Except during manipulation, conditioning, or testing, samples shall be stored in their original containers, bags, etc., that were supplied by the user. Facility-supplied samples shall be stored in dust-free and noncontaminating containers that do not contact the sample test surface (e.g., polypropylene wafer shippers) and that have been cleaned in the same manner as the witness samples.

6. TEST PROCEDURES

General procedures

6.1 Labeling. Sample containers and bags shall be clearly labeled. Sample containers consisting of more than one part shall be labeled on each part. If possible, each individual sample shall be labeled in a manner that uniquely identifies the sample and the side to be exposed.

6.2 Witness samples. A witness sample, placed at normal incidence to the direction of O-atom attack (or in the harsh O-atom environment in a Level 1 facility) and maintained at 25-30°C, shall be used to verify the effective fluence to which the test samples are exposed {11,12}. Kapton HN is the recommended witness sample, although a carbon film from York University (see Section 4.6) may be used if its erosion rate has been measured relative to that of Kapton HN in the same facility. Even if there is an independent method of measuring fluence, a Kapton HN witness (or a carbon witness that can serve as a measure of Kapton-equivalent fluence) shall be used. If the exposure area is small and sample exposure requirements are such that a Kapton HN witness cannot be exposed simultaneously, then the exposures of the test and witness samples shall be done in series, while maintaining as closely as possible the same conditions for both exposures. If many samples are to be exposed in series under the same set of conditions, a witness sample shall be exposed at the beginning, middle, and end of the series. (The witness control, if used, shall be changed with each change of a witness sample.) If the exposure area is large enough to accommodate many test samples, then witness samples shall be placed such that the uniformity of the whole exposure area will be checked. In plasma facilities, witness samples shall be placed as close as possible to the same axial and radial locations as the test sample, in order to ensure minimization of errors in the effective atomic oxygen fluence. If similarity of orientation cannot be achieved, then axial, radial, and orientational erosion rate characterization of the plasma chamber shall be performed to allow prediction of the relative atomic oxygen fluences at the positions of the test and witness samples.

6.3 Control samples. In general, a control sample shall accompany each test or witness sample through all stages of handling, testing, and analysis. For witness samples to be weighed, every witness sample that is subjected to direct O-atom attack shall have a corresponding, identical control sample in the chamber, and the control sample's test surface shall face away from the harsh O-atom environment. In Level 2 and 3 facilities, the witness sample and its control shall be mounted back-to-back with the witness facing the beam and the control facing away. In a plasma facility, the control sample shall not be in contact with the plasma and shall face away from it. If a recession measurement of witness samples is specified, then witness controls are unnecessary. Control-sample strategy may vary depending on the nature of the tested material. The evaluation of a protective coating on a substrate shall be accompanied by evaluation of the substrate material without the coating. For most other materials, the control samples shall be placed in the exposure chamber but out of direct atomic oxygen attack. The

control samples should be subjected to the same functional tests as the test samples. If it is suspected that a surface oxide from reaction with thermal O atoms could affect a functional test, then the sensitive control samples shall remain in their containers outside the test chamber during exposure.

6.4 Constant vs. incremental exposure. Constant exposure of the test samples until the desired fluence is reached is recommended. Any test that is likely to be or has been interrupted for any reason shall only be accepted if the user gives pre- or post-test approval {13}.

6.5 Chamber pressure. If it is assumed that the background gases are mainly components of air, the average pressure in a beam-based facility during a test is not critical, as long as the mean free path is long compared to the chamber dimensions. This condition shall be considered to be satisfied at pressures below 10^{-4} Torr. The operating pressure in a plasma- or flow-cell-based facility shall be kept above 60 mTorr to minimize backstreaming of roughing pump oil. To minimize outgassing contributions of adsorbed gases to a plasma, a plasma chamber shall be evacuated to a pressure of less than 150 mTorr for at least 30 minutes prior to initiation of the plasma.

Evaluation of exposure fluence {14,15}

6.6 Weight-loss measurement. If weight-loss is specified as the method to verify exposure fluence, then the facility operator is responsible for performing the verification measurements. Witness samples shall be weighed before and after a test. Before weighing, the samples shall be conditioned in vacuum to account for possible moisture absorption. Although a standard analytical balance may be used for weighing, a microbalance will produce more precise and, in principle, more accurate results {16}. The balance that is used shall be capable of a precision of ≤ 5 percent in the measurement of the expected weight loss of the test-fluence witness sample(s).

6.6.1 Before exposure, fresh witness samples and their controls shall be placed in a clean vacuum chamber and allowed to outgas at ambient temperature in a low pressure environment of $\leq 10^{-5}$ Torr for 8 or more hours. The samples shall be removed from their original containers and placed on a clean surface or in a clean mount that has been cleaned with solvent in a manner similar to that described in Section 5.3. The outgassing hardware need not be cleaned before every conditioning run if it has been stored in the vacuum chamber or in another clean environment. The sample side that is to be tested shall not be in contact with a surface during outgassing. A mark on each sample that would identify it and indicate the back side is recommended.

6.6.2 After vacuum conditioning, the chamber shall be back-filled with dry nitrogen or argon. The samples shall be removed from the chamber, and within five minutes, they shall be placed in their original containers (without lids or with the lids loosely attached) or on a clean surface and then sealed for a minimum of 15 minutes in a desiccator which has fresh desiccant.

6.6.3 Each sample shall be removed from the desiccator and weighed individually. A stopwatch shall be started the moment the desiccator seal is broken, and the sample weight shall

be recorded at one-minute intervals for at least four minutes. The true, or "dry" weight of the sample is determined by extrapolation back to the weight at the time the sample was removed from the desiccator.

6.6.4 After the sample is weighed, it may be sealed in its original container for storage outside the desiccator.

6.6.5 After the test is complete, the samples shall again be conditioned for 8 or more hours under vacuum, or they shall remain in their mounts in the vacuum chamber in which the exposure was carried out until a minimum of 8 hours (exposure time plus conditioning) have elapsed. The samples are then removed from the vacuum chamber and weighed, as described in Section 6.6.3.

6.6.6 Weight loss (or gain) of each witness sample shall be corrected by the amount of weight loss or gain in the control sample.

6.6.7 The corrected weight loss is used in the following formula to determine the effective test fluence:

$$F_{\text{test}} = \Delta M_w / (A_w \rho_w E_w)$$

where

$$F_{\text{test}} = \text{fluence of test, atoms/cm}^2$$

$$\Delta M_w = \text{weight loss of witness sample, g}$$

$$A_w = \text{surface area of witness sample exposed to O-atom attack, cm}^2$$

$$\rho_w = \text{density of the witness sample, g/cm}^3$$

$$(\rho_{\text{Kapton HN}} = 1.42 \text{ g/cm}^3)$$

$$E_w = \text{erosion yield of witness sample, cm}^3/\text{atom}$$

$$(E_{\text{Kapton HN @ 298 K}} \equiv 3.00 \times 10^{-24} \text{ cm}^3/\text{atom})$$

6.6.8 To find the erosion yield of a test sample (designated by the subscript s), the effective fluence is used:

$$E_s = \Delta M_s / (A_s \rho_s F_{\text{test}}) = E_w \Delta M_s A_w \rho_w / (\Delta M_w A_s \rho_s)$$

6.7 Recession measurement. The exposure fluence of a test may be determined by a step-height measurement of a witness sample, which may be performed by the facility operator or the user. In the second case, verification of exposure fluence is entirely the user's responsibility. If a step-height measurement is to be made, vacuum conditioning and weighing are unnecessary. The simplest method of measuring a step height is profilometry with a stylus profilometer. Other techniques, e.g., atomic force microscopy (AFM), optical interferometry, and scanning electron microscopy (SEM), may be used. Whichever method is used, the apparatus shall be calibrated periodically with known standards to ensure accurate measurements {17,18}, and the calibration shall be kept on file and provided to the user upon request.

6.7.1 A simple metal mask may be cut from a sheet of stainless steel or nickel etched mesh. This mask shall be cleaned in the same manner as the sample and then mounted or clamped in direct contact with the sample. The mesh provides for many steps over the whole sample surface, so several steps shall be measured and averaged. Such a mask will work well for beam sources that do not have high radiative heat loads. The user shall discuss possible mask heating with the facility operator before specifying a metal mask {19}.

6.7.2 The following guidelines shall be used for mesh dimensions {20}. (Areal dimensions for other mask types may be inferred from these dimensions.)

<u>test</u>	<u>width of square opening</u>	<u>width of wire</u>	<u>thickness</u>
directed beam	> 0.020"	≥ 0.005"	≤ 0.004"
plasma	≥ 0.040"	0.010" (per 10 ²⁰ atoms/cm ² effective fluence)	≤ 0.004"

6.7.3 A variety of coatings may serve as effective masks, including metals, SiO₂, and Al₂O₃. To ensure protection of the underlying material, these coatings shall be ≥ 20 nm thick. If a coating is applied to the surface, then its thickness must be measured because the total step height will include the coating thickness. A simple, nonmetal coating may be formed by lightly spraying a saturated sodium chloride/water solution on the sample and allowing it to dry. If the spray is sparse, then salt islands will be left on the surface, and these islands will protect the material underneath. After exposure, the salt may be dissolved away by rinsing the sample in water; then the step-height measurement will not include the coating thickness {21}.

6.7.4 The effective atomic oxygen exposure fluence is calculated from a recession measurement as follows:

$$F = S_w/E_w$$

where

S_w = step height, or erosion depth, cm

E_w = erosion yield of witness sample, cm³/atom

7. SPECIFICATION OF TEST PARAMETERS

For each test, the user shall specify the test parameters according to the guidelines below.

7.1 Level of testing. The classification level (and sub-level, if applicable) of the test to be performed shall be specified.

7.2 Number of samples. The number of samples to be tested, the number of exposure batches, and the sample makeup of each batch shall be specified {22}.

7.3 Orientation (applies to Level 2 and 3 tests only). The orientation of the samples to be tested with respect to the direction of O-atom attack shall be specified.

7.4 Sample temperature. The temperature at which the samples are to be maintained during a test shall be specified {23}.

7.5 Contamination. The nature and allowable levels of various contaminants that accumulate on the witness samples may be specified. For example, the maximum allowable atom percent or number of monolayers of certain species may be specified. If atom percent is to be specified, then the user shall specify the method by which the atom percentages will be determined {24}. If the contamination level is specified, then the individual responsible for its verification and the method of evaluation shall be indicated. If the facility operator is responsible, then the measured contamination levels shall be included in the test report (Section 8) under the heading of "Special requests."

7.6 UV/VUV light (applies to Level 3 tests only). The solar-equivalent UV/VUV exposure level and spectrum, if applicable, shall be specified.

7.7 Ions (applies to Level 3 tests only). The identity and flux of charged particles that accompany an exposure to ground-state atomic oxygen shall be specified.

7.8 Witness samples. The type and number of witness samples to be used for a test and any deviations from standard witness sample procedure (Section 5.2) shall be specified.

7.9 Atomic oxygen fluence. The O-atom fluence for a test shall be specified in units of atoms/cm². Unless otherwise specified, the acceptable measured deviation from the requested fluence is ± 20 percent. The specified fluence shall refer to the "Kapton-equivalent fluence," as defined by the erosion of a Kapton HN witness standard whose erosion rate is assumed to be 3.00×10^{-24} cm³/atom at 25°C and whose density is taken to be 1.42 g/cm³ {25}.

7.10 Atomic oxygen flux. The O-atom flux for a test, based on a Kapton HN standard, shall be specified in units of atoms/cm²/s {26}.

7.11 Analysis methods. The method of analysis (i.e., weight loss or recession) to be used to verify the exposure fluence of a test shall be specified. If applicable, other instructions to the facility operator on *in* or *ex situ* analysis of test, witness, or control samples shall be given.

7.12 Constant vs. incremental exposure. If applicable, instructions to the facility operator on the exposure increment shall be given {27}.

7.13 Disposition of samples. The post-test disposition of all samples, including test, witness, and control samples, shall be specified.

7.14 Special Instructions. Any additional instructions regarding the test shall be given under this heading.

8. TEST REPORT

The facility operator shall prepare a report for each test with contents outlined as follows:

8.1 Sample log. Log of samples received, by code number or name, including witness samples.

8.2 Sample handling. Techniques and details of pre- and post-test handling, conditioning, and cleaning (as applicable).

8.3 Sample mounting. Mounting configuration and locations for test samples, witnesses, and controls; for multiple exposures, composition of batches and/or order of exposure. Diagrams are recommended.

8.4 Sample temperature. Measured temperature, or temperature range, of test samples, witnesses, and controls. If temperature was not measured, then the estimated temperature of the test samples during the test shall be given.

8.5 Exposure duration. Exposure time in minutes (and number of pulses, if applicable) of test samples to the O-atom beam or harsh O-atom environment.

8.6 Exposure fluence. Description of analysis used to evaluate 5 eV Kapton-equivalent O-atom exposure fluence, including raw data and estimated uncertainties.

8.7 Kinetic energy (applies to Level 2 and 3 tests only). Nominal O-atom kinetic energy during exposure; description of how it was measured or estimated for the test.

8.8 Special conditions. Departures from facility qualification document descriptions of the typical exposure environment.

8.9 In situ analysis. Description of procedures used for *in situ* analysis, if applicable.

8.10 Synergistic exposure environment (applies to Level 3 test only). Flux, fluence of VUV light and/or ions that accompanied the atomic oxygen exposure; identity and kinetic energies of ions.

8.11 Special requests. Response to special requests by user.

9. REPORT ON GROUND-SPACE CORRELATION

A brief report, prepared by the user, comparing the results of the ground-based test with results from space-based experiments on the same materials, is optional {28}.

NOTES

1. Exposure parameters are to be based on experimentally measured quantities. When experimental measurements are especially difficult or uncertain, upper or lower bounds should be determined by the sensitivity limits of the attempted measurements. Only as a last resort should parameters be quoted as estimates based on theoretical calculations alone.
2. The contamination level in the exposure environment or in the chamber may be checked with the use of a nonerodible witness sample, such as magnesium fluoride, silicon dioxide, or germanium-coated Kapton HN.
3. The accumulation of contamination on an erodible surface is generally a fraction (perhaps one quarter) of that on a nonerodible material.
4. Fluorine contamination may be a problem in a facility that typically yields acceptably low contamination levels. Some atomic oxygen sources produce seemingly negligible amounts of atomic fluorine. On certain materials, usually inorganic, fluorine may bind more preferentially than oxygen with atoms on the surface of the test material, giving rise to anomalously high surface fluorine concentrations which grow with exposure fluence.
5. The control samples are distinct from witness samples, as they are not subjected to direct O-atom attack. Control samples can be witness or test sample controls.
6. Silicon contamination will invalidate a test by reacting with O atoms to form a non-reactive SiO_x layer.
7. No system should use air, which, because it contains water and nitrogen, could lead to production of atomic hydrogen, metastable nitrogen, and nitrogen oxide compounds.
8. The maximum fluence attainable may be much higher than that reported if the test can be interrupted for required facility maintenance. Ultimately, the length of a test will be limited by the buildup of contamination.
9. Good and bad correlations should be included to allow the user to judge the potential of obtaining reliable results for the particular material to be tested.

10. Cans of freon or compressed gas are a potential source of contamination and should not be used to remove dust from samples. For certain coatings to be tested, neither the test, witness, nor control samples shall be cleaned. Avoiding cleaning is particularly important for a coating to be evaluated *in situ* with the use of a quartz crystal microbalance whose calibration has already been determined.
11. Given ample space, a nonerodible, contamination witness sample should also be placed near the test sample(s) where it can sample the harsh O-atom environment.
12. Oxidation or outgassing products from test samples might affect the erosion rate of a witness sample. Such problems of contamination on a witness sample can be detected by comparing the effective fluence, derived from the witness sample, with that from an exposure operated under the same conditions with only the witness sample present.
13. Special circumstances requiring interruption of a test and exposure of the samples to air before the test is complete may be unavoidable if facility maintenance is needed or if the progression of degradation is to be studied by some technique that must be utilized outside the chamber. Such interruptions should be minimized because transient reaction products residing on the sample surface may react with air and alter the surface chemistry. Also, any interruption increases the risk of contamination.
14. The erosion yield of a Kapton HN witness sample (or a secondary standard witness sample) must be determined in order to evaluate the effective (Kapton-equivalent) fluence of a test. The procedures described here apply equally well to erosion yield measurements on test samples. Erosion yield measurements can be separated into two groups: mass loss and recession. Mass-loss measurements can be carried out *in situ* if the sample material is deposited on the crystal of a quartz crystal microbalance or *ex situ* by weighing the sample on a balance. Only weight loss and recession measurements will be considered in this protocol for the evaluation of exposure fluence. Weight loss measurements must be performed very carefully to avoid errors associated with absorption of moisture. Furthermore, an accurate weight-loss measurement might be impossible if the sample is crumbly and can lose particles. Recession, or step-height, measurements obviate the need for carefully controlled weight-loss measurements, but they require proper masking and generation of a step whose height can be measured accurately. Also, the step-height method may fail if the eroded surface is not smooth relative to the size of the step (i.e., when the exposure was not long enough to yield approximately 300 nm of erosion or greater). Weight-loss measurements are best performed on site by the facility operator, whereas recession measurements can be carried out with equal effort on site or in the user's laboratory. Done properly, either method to determine erosion yield is valid. It should be noted, however, that weight loss and step height methods are of limited value for assessing materials of low reactivity (e.g., protective coatings). The *in situ* method employing a quartz crystal microbalance may be the only precise way of accurately measuring the erosion yields of relatively unreactive materials. The method used to verify the exposure test fluence should be decided through discussions with the facility operator.

15. The erosion of a material is likely not to be truly linearly dependent on the O-atom fluence. For example, Tennyson {Can. J. Phys. **69**, 1190 (1991)} has observed an initial induction period for some materials, after which the erosion rate increases and becomes linear. In addition, Bruce Banks {private communication} has suggested that in long exposures of Kapton HN, particulate impurities inside the polymer that are resistant to O-atom attack build up on the surface and cause the erosion rate to decline at high fluences ($> 10^{21}$ atoms/cm²). In light of these findings, the true fluence of a test will actually be higher than that derived from a Kapton HN witness sample. However, the error in the test fluence will be negligible for fluences between 10^{20} and 10^{21} atoms/cm². Even above 10^{21} atoms/cm², the Kapton-equivalent fluence should be within 20 percent of the true value.
16. A typical test fluence of 2×10^{20} atoms/cm² will lead to a mass loss of ~ 0.001 g in a 1/2" diameter Kapton HN witness sample. This small change in mass is difficult to measure accurately with an analytical balance.
17. As a general rule, the facility operator shall only be responsible for providing a masking method for the witness samples. The user shall be responsible for providing a mask or masking method for test samples, unless other arrangements are made with the facility operator.
18. The user should exercise caution when choosing samples on which to perform recession measurements. The stylus profilometer can plow through a step in a soft material, such as carbon; AFM is limited in the area that it can image, so AFM might fail to reveal a picture of the entire step if it occurs over a distance of ~ 100 μ m or greater; an imaging optical interferometer might be unable to measure a step height if the difference in reflectance between the eroded and noneroded regions is high; and SEM requires that the sample be cleaved or cut so the cross section of the step can be viewed.
19. Beam sources that produce large quantities of light (> 1 sun at the sample surface) may locally heat the mesh to high enough temperatures to melt or otherwise damage the surface of the underlying material, even when the sample is in a cooled mount. A metal mask can also be heated inductively in a plasma system, although the ultimate temperature to which the mask might rise is not necessarily enough to preclude its use in a test.
20. The size of the masked area is important if the atomic oxygen exposure can lead to undercutting of the mask. Complete undercutting of the mask will invalidate a step-height measurement. Undercutting is usually only a problem in thermal-atom facilities, where the angle of O-atom impingement is not well-defined.
21. The disadvantage of the salt mask is that it is irregular. Depending on the size of the uneroded areas, a stylus profilometer might not be usable, and another, more costly, step-height measurement would be necessary.

22. Possible effects of cross-contamination should be taken into account when one is choosing the samples that are to be exposed simultaneously. For example, a sample that could outgas silicone might minimize degradation of a neighboring sample by leading to the formation of a protective SiO_x layer.
23. This temperature is a bulk temperature and is based on the measured temperature of the sample mount. The actual surface temperature of the sample will depend on the sample's thermal properties and how it is mounted. Also note that extreme temperatures could lead to cracking of coatings on coated samples and thereby open up sites for enhanced atomic oxygen erosion.
24. It should be borne in mind that the takeoff angle in an XPS measurement will affect the measured atom percent.
25. The minimum fluence that should generally be used for a test is 5×10^{19} atoms/cm².
26. Typical fluxes available and acceptable with current test facilities range between 10^{14} and 10^{17} atoms/cm²/s.
27. Constant exposure for the duration of the test is recommended, unless special circumstances, such as the need for a particularly high fluence or for studies of progressive degradation, require the interruption of the exposure.
28. Users frequently either test materials in space or have access to space data. If the user has space data on materials that have been tested in a ground-based facility, the data and perhaps a description of the space-based experiment may as a courtesy be provided to the facility operator. As mentioned earlier, the validity of a test rests on the ability of the facility to produce results that can in a straightforward manner be applied to the prediction of behavior in LEO. The augmentation of a facility's database on the correlation between ground- and space-based tests will serve the space environmental effects community well in the effort to carry out valid atomic oxygen testing in ground-based facilities.